

TECHNICAL REPORT ARCCB-TR-01003**CHARACTERIZATION OF TANTALUM LINERS
APPLIED TO 25-MM AND 120-MM CANNON
BORE SECTIONS VIA EXPLOSIVE BONDING**

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13. ABSTRACT (Maximum 200 words) A materials forensics analysis was performed on sections of 25-mm rifled and 120-mm smoothbore gun tubes that were explosively bonded with pure tantalum. Characterization tests included liner thickness measurements, profilometry/surface finish, x-ray diffraction, and microstructural analysis including microhardness, scanning electron microscopy, energy dispersive spectroscopy, and adhesion testing. Results indicate that there was good adhesion. However, there was evidence that the explosive bonding process was overly energetic; both adiabatic shear bands in the steel and a brittle iron/tantalum intermetallic phase in the coating were observed.					
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BACKGROUND

The internal surface of a 25-mm rifled cylinder and a 120-mm smoothbore cylinder were explosively bonded with tantalum. This work was conducted by Technology to Products on the Leading Edge (TPL) as part of an Army Research Office (ARO) Small Business Innovative Research (SBIR), Phase II effort. Benet Laboratories received a sample from each of these bonded tubes (each specimen received was a half cylinder approximately one-inch in length) for characterization in order to determine the properties/integrity of the liner and substrate. The results of these analyses are described below.

APPROACH

The samples provided were characterized using an established protocol. Benet has been using this protocol to evaluate its sputtered coatings, as well as the coatings/liners provided by other organizations involved with bore protection technologies. The specific characterizations performed on the explosively bonded samples were:

- Liner thickness
- Profilometry/surface finish
- X-ray diffraction
- Microstructural analysis including microhardness
- Scanning electron microscopy (SEM) and energy dispersive spectroscopy (EDS)
- Adhesion

The results of these tests are described below.

LINER THICKNESS

25-mm Specimen

Upon stereoscopic observation, it was evident that the tantalum liner did not conform to the rifling profile (Figure 1). The thickness of the tantalum liner on the 25-mm specimen was approximately 1-mm or 0.040-inch (see Figure 2). Note that the dark features in the tantalum are artifacts left from the polishing. Polishing artifacts were embedded in the very soft liner material.

120-mm Specimen

The liner thickness of the 120-mm sample was measured using an optical microscope. As can be seen in Figure 3, the coating is approximately 635- μ m (0.025-inch) thick. The dark features in the coating are artifacts left from the polishing. Polishing artifacts were embedded in the coating because it was so soft. For the 120-mm sample, a feature was observed on the surface of the liner that appeared to be in the shape of a weldment. It is believed that a welded tantalum tube was used for this process and that the weldment profile was transferred to the surface of the liner. This feature would require subsequent machining of the liner.

PROFILOMETRY/SURFACE FINISH

120-mm Specimen

Surface profiling and finish measurements were performed using a Federal® SURFANALYZER® SYSTEM 2000, currently in use by the Watervliet Arsenal (WVA) for determining surface finish of electroplated chromium on cannon bores. This is a stylus instrument that employs a 10- μ m radius spherical tracer head to obtain displacement data. Surface finish was determined as specified in the standard ANSI/ASME B46.1-1985, Surface Texture. Per this specification, all surface finishes reported here are the arithmetic average of the absolute values of the measured height deviations from the centerline along the surface of each specimen.

Table 1 presents "peak-to-valley" height deviations and surface finish for both an area located a significant distance away from the apparent weldment and for the weldment itself. For comparative purposes, these values are also reported for a cylindrically magnetron sputtered (CMS) tantalum coating (Specimen #CMS-Kr6-S2-00504).

Table 1. Surface Profilometry of Explosively Bonded Versus Sputtered Tantalum Coatings

Specimen	Peak-to-Valley Distance (microinches)	Surface Finish (microinches)
120-mm	550	50 to 80
120-mm @ Weld Seam	22.4×10^3	90 to 120
CMS-Kr6-S2-00504	75	16

The current specifications adopted by WVA for electrodeposited chromium on smoothbore 120-mm cannons require a surface finish no greater than 16 microinches. In practice, surface finishes of electroplated high contraction chromium on smoothbore 120-mm tubes plated at WVA typically do not exceed eight microinches (ref 1).

From simple visual inspection of the explosively bonded specimen, it was obvious that the peak-to-valley distance (height deviation) of the weld seam most likely exceeded the specifications for any medium or large caliber gun bore. Profilometry indicated that the height of the weld seam was 0.0224 inch. It has recently been reported that the contractor intends to eliminate the presence of this seam by explosively bonding a preformed tube of tantalum to the steel substrate (as opposed to explosively bonding a shaped and welded sheet).

However, results presented in Table 1 also show that at a location well away from the weld seam, the explosively bonded coating has a surface finish of 50 to 80 microinches, exceeding the current specifications for the smoothbore 120-mm by more than a factor of three. This implies that utilization of the explosively bonded method in its current state will require an additional machining step (e.g., honing) in order to meet current specifications for the 120-mm cannon. In contrast, it was found that Benet's CMS tantalum coating in the as-deposited state satisfies the current surface finish specifications for the smoothbore 120-mm.

X-RAY

25-mm Specimen

Scans were taken using copper (line source) at 40 kV and 40 mA. A 2-mm round collimator was used to limit beam area of the sampling surface. Collimator slits were 0.5- and 1-mm on the detector side, and 4- and 2-mm on the x-ray beam side. Soller slits were used for both the detector and the x-ray source for beam conditioning. Scans were performed at a resolution of 0.1 degree at 1.0 second and 1.5 seconds per point from 5° to 145° two-theta.

The 25-mm specimen showed a blackish-grayish color, and the surface was very rough. The color of the liner material was also uneven. The results of the x-ray analysis are listed in Table 2. Some texture runs were conducted for the 25-mm sample. They showed poles, which represent some azimuth asymmetry. Further analysis and interpretation of the texture data are being conducted.

Table 2. X-Ray Diffraction Results on 120-mm and 25-mm Specimens

Specimen	Surface Phase	Preferred Orientations	Other Observations
120-mm #1	α -Phase Tantalum with Tantalum Oxide (high intensity run)	α -Tantalum; Preferred (200)	Grayish-blackish non-uniform coatings
120-mm #2	α -Phase Tantalum	α -Tantalum; Preferred (200)	Shining areas with horizontal polishing marks
25-mm	α -Phase Tantalum with Tantalum Oxide (high intensity run)	α -Tantalum; Preferred (200)	Specimen showed very uneven surfaces. The color was also uneven, showing areas of grayish-blackish color; some areas seem exposed.

120-mm Specimen

The identical procedure was followed with the 120-mm sample as with the 25-mm sample. The 120-mm sample showed distinctively two areas, and the surfaces were relatively smoother compared to the surfaces of the 25-mm sample. The results of the x-ray analysis are listed in Table 2. Some texture runs were conducted for the 120-mm sample. They showed poles, which represent some azimuth asymmetry. Further analysis and interpretation of the texture data are being conducted.

MICROSTRUCTURAL ANALYSIS

25-mm Specimen

Microstructure of the Steel Substrate

The microstructure of the steel substrate consisted of a quenched and tempered martensitic microstructure, as is typically observed in gun tubes (Figure 4). The rifling of the gun tube appeared to be rounded, suggesting prior firing damage in this gun tube. However, some medium caliber gun tubes can contain "gentler" rifling profiles. Additionally, there were cracks on one side of a land (Figure 5). The contractor later confirmed that, in fact, the gun tube had previously been fired.

Plastic flow lines, due to the explosive bonding process, were observed in the steel. Also, a few isolated areas of what appears to be adiabatic shear were observed (Figure 6). Adiabatic shear occurs during dynamic loading. During dynamic loading, localized deformation and heating occurs along shear bands. In steels this localized heating can cause the formation of untempered martensite (refs 2,3). Untempered martensite is never desirable in a gun tube, being extremely brittle and adversely affecting fatigue life. However, without further testing, we are not able to quantify this effect. Adiabatic shear has been observed after in-bore detonation of gun tubes (ref 3), but not during normal gun firing.

A heat-affected zone appears to have formed in some regions of the steel substrate (Figure 7). However, the microhardness in this zone was slightly lower than that in the base steel (Table 3). The softening in this zone could be due to overtempering or to redistribution of solute elements from the steel into the intermetallic phase at the interface.

Table 3. Knoop Microhardness Results for 25-mm and 120-mm Specimens

Location	Hardness in 25-mm	Hardness in 120-mm
Liner	HK ₃₀₀ 145 (~HRB 73)	HK ₃₀₀ 198 (~HRB 89)
Interfacial Phase	HK ₂₅ 742 (~HRC 60)	Not Measured
Gun Steel	HK ₃₀₀ 373 (~HRC 37)	HK ₃₀₀ 264 - HK ₃₀₀ 421 (~HRC 23 to 42)
Heat-Affected Zone in Steel	HK ₂₅ 337 (~HRC 34)	Not Measured

Microstructure of the Interface

An intermetallic layer (Figures 6 through 8) was observed along the majority of the liner/substrate interface in the 25-mm specimen. The intermetallic layer was thicker in the 25-mm sample than in the 120-mm sample. In the thickest regions of the 25-mm specimen, the layer was approximately 65- μ m (0.0025 inch) thick. Additionally, in many areas of this layer

there were cracks observed (see Figure 8). The hardness of this layer was much harder than both the steel and the liner itself. An EDS spectra later confirmed this layer to be a tantalum/iron intermetallic compound (see "SEM and EDS" section); therefore, *melting must have occurred at the interface*. Voids were also present along the interface (Figures 8 and 9). Many of these voids appeared to be on a preferential side of the "wavy" interface.

According to the literature, explosive bonding can be achieved without melting and intermetallic compound formation. It is only when the process is too energetic that such undesirable features are formed (ref 4). If the pressurization can be controlled, then undesirable features such as these may be mitigated.

Microhardness of the Liner

The microstructure of the coating contained embedded polishing debris, while the gun steel substrate did not; therefore, the liner material was softer than the substrate. This was verified by microhardness readings (Table 3). The low microhardness readings in the liner correlate with the ultimate tensile strength of pure tantalum (refs 5,6). The liner material appeared to be a single-phase, and was dense and defect-free.

120-mm Specimen

Microstructure of the Steel Substrate

The microstructure of the steel substrate consisted of both fine and coarse pearlite and ferrite. This microstructure suggests that the steel was in the normalized condition when explosively bonded (Figure 10). The microstructure of the steel near the interface was "pancaked" due to plastic deformation induced by the explosive bonding process (Figure 11). As with the 25-mm sample, there was evidence of adiabatic shear in the 120-mm sample (Figure 12).

Microstructure of the Interface

As with the 25-mm sample, the interface of the 120-mm sample also contained an interfacial layer. Although this layer in the 120-mm sample was not analyzed by EDS, it is believed to consist of the same iron/tantalum intermetallic as that observed with the 25-mm sample. The intermetallic was less thick in the 120-mm sample ($< 25\text{-}\mu\text{m}$ thick; $< 0.001\text{-inch}$) than in the 25-mm sample. Much of the intermetallic contained cracks and voids (Figures 13 and 14). Additionally, much of the intermetallic was detached from the interface and present as "islands" in the tantalum coating. No voids were observed at the coating/steel interface.

Microstructure of the Liner

The microstructure of the liner material contained embedded polishing debris, while the gun steel substrate did not; therefore, the liner material was softer than the substrate. This was verified by microhardness readings (Table 3). The low microhardness readings in the liner

correlate with the ultimate tensile strength of pure tantalum (refs 5,6). The liner appeared to be a single-phase, and was dense and defect-free.

SEM AND EDS

25-mm Specimen

Figure 15 shows the results of SEM and EDS performed on the 25-mm specimen. Tantalum was observed in the liner (regions 1 and 4), tantalum and iron were observed in the intermetallic (region 2), and iron, along with alloying elements of gun steel, was observed in the steel (region 3). In region 2, the relative intensities of tantalum and iron in the intermetallic suggest that it is probably iron/tantalum (ref 7).

ADHESION

Groove and impact testing were performed in compliance with ASTM Designation B571-97, "Standard Test Methods for Adhesion of Metallic Coatings." Groove testing was performed using a Southbend™ Shaper Model groove test apparatus with tungsten carbide tip. Impact testing was performed using the WVA Quality Assurance Office's Brinell hardness testing apparatus. All images were acquired using the 1LM21W Nikon/Lasertech Laser Scanning Microscope (LSCM) in two-dimensional extended depth of field or "optical" mode at a magnification of 200X.

25-mm Specimen

Due to the limiting size of this specimen, a second, perpendicular groove was not cut. No lifting or tearing of the liner material was evident along any of the edges of the imparted groove. It is important to consider the softness (Knoop hardness ~150) of this liner material when interpreting these results. Groove testing depends on a hard coating for appropriate test severity. Similarly, when performing impact testing on a soft coating, there will not likely be evidence of spallation or cracking in the coating. Given the softness of the liner, we do not feel that groove or impact testing was suitable as a discriminating measure of adhesion.

120-mm Specimen

No lifting or tearing of the coating was seen along any of the edges of the imparted grooves. Similarly, results from impact testing of this specimen indicated an extremely ductile coating, with no cracking detected within the resultant indent or in the immediate area surrounding it. Given the softness of the liner, we do not feel that groove or impact testing was suitable as a discriminating measure of adhesion.

SUMMARY OF RESULTS

25-mm Sample

- The liner was approximately 1-mm thick and did not conform to the rifling profile.
- The steel microstructure was tempered martensite. Plastic deformation, adiabatic shear, and a heat-affected zone occurred in the steel from the explosive bonding process.
- Along most of the interface, a brittle iron/tantalum intermetallic compound containing cracks and voids was observed by microscopy, microhardness, and EDS. These features were created by an overly energetic explosive bonding process.
- The liner was pure tantalum as determined by x-ray diffraction and microhardness.
- Adhesion was good; however, tests were inconclusive because of the extremely soft liner hardness.

120-mm Sample

- The liner was approximately 635- μ m thick and contained what appeared to be the profile of a weldment on the surface of the liner. The surface finish of the liner was 50 to 80 microinches and did not meet the specifications for a 120-mm gun tube.
- The steel microstructure was a mixture of ferrite and pearlite (normalized condition). Plastic deformation, adiabatic shear, and a heat-affected zone formed in the steel due to the explosive bonding process.
- Iron/tantalum intermetallic "islands" containing cracks and voids were observed along much of the coating interface. These features were created by an overly energetic explosive bonding process.
- The liner was pure tantalum as determined by x-ray diffraction and microhardness.
- Adhesion was good; however, tests were inconclusive because of the extremely soft liner hardness.

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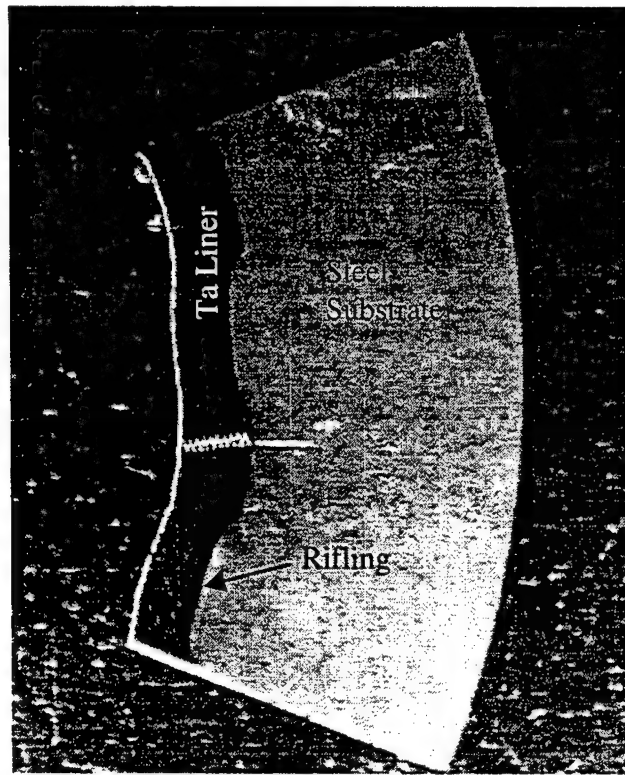


Figure 1. Stereophotomicrograph showing tantalum liner on 25-mm specimen. Note that the coating did not conform to the rifling profile.

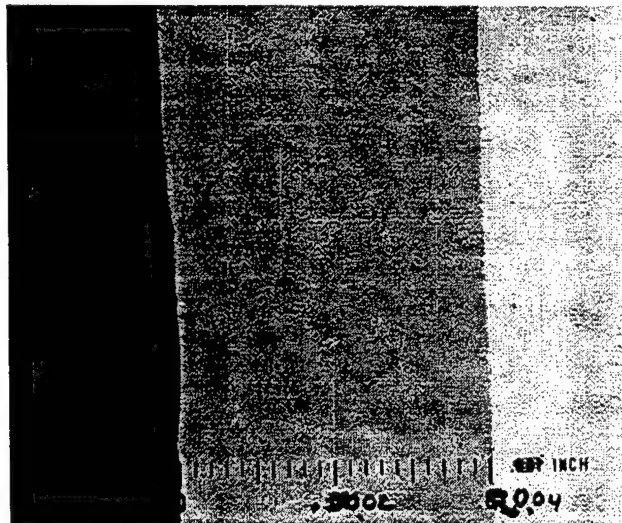


Figure 2. Optical photomicrograph showing liner thickness of approximately 1-mm (0.040-inch) on the 25-mm specimen.

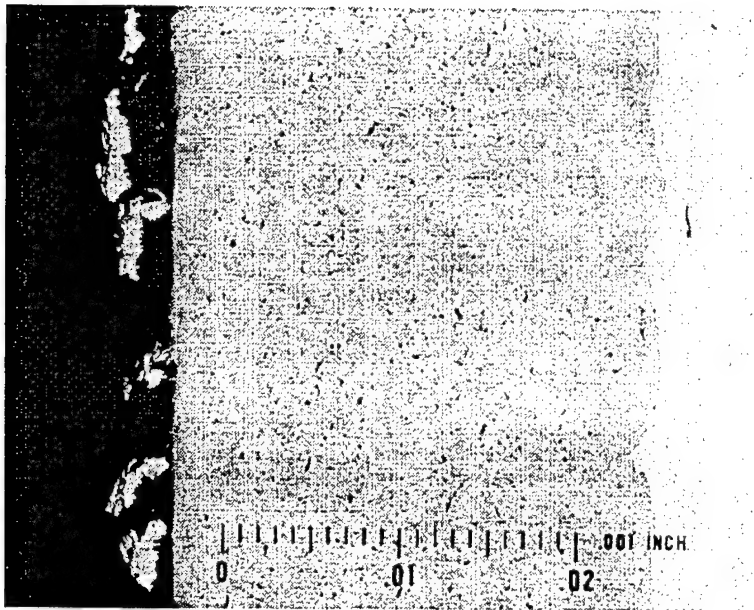


Figure 3. Optical photomicrograph showing coating thickness of approximately 635- μm (0.025-inch) on the 120-mm specimen.

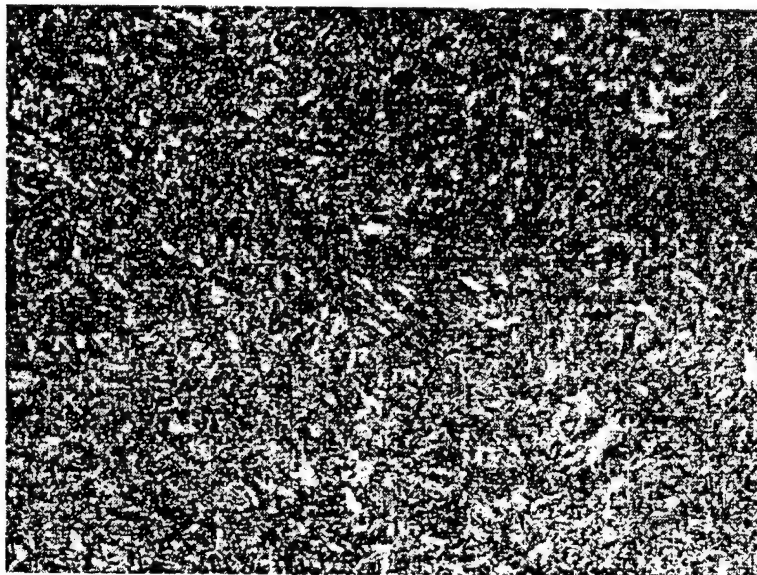


Figure 4. Optical photomicrograph showing tempered martensitic microstructure of the steel in the 25-mm specimen (1000X).

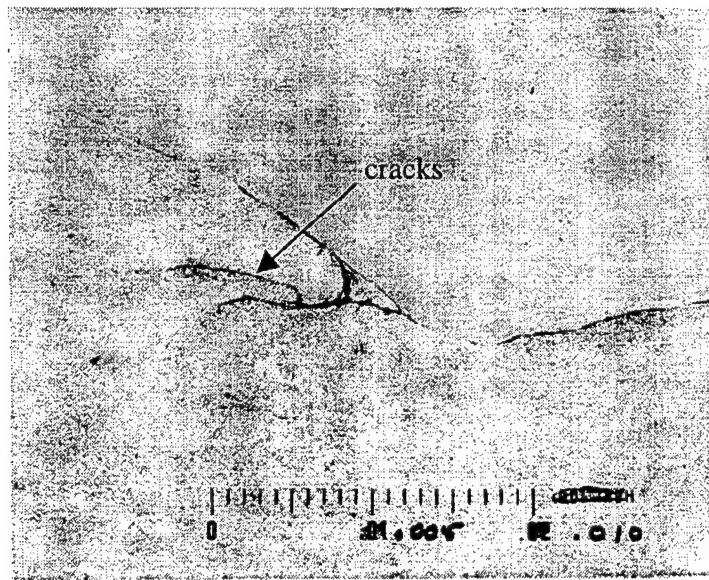


Figure 5. Optical photomicrograph showing cracks in the side of a land (200X).

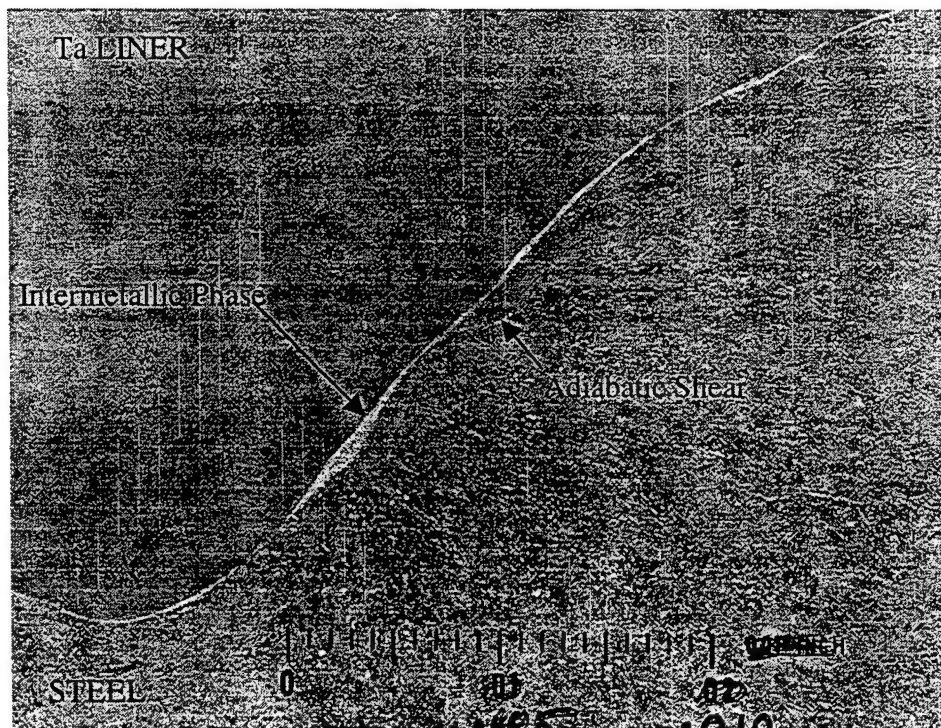


Figure 6. Optical photomicrograph showing intermetallic phase at interface and adiabatic shear (200X).

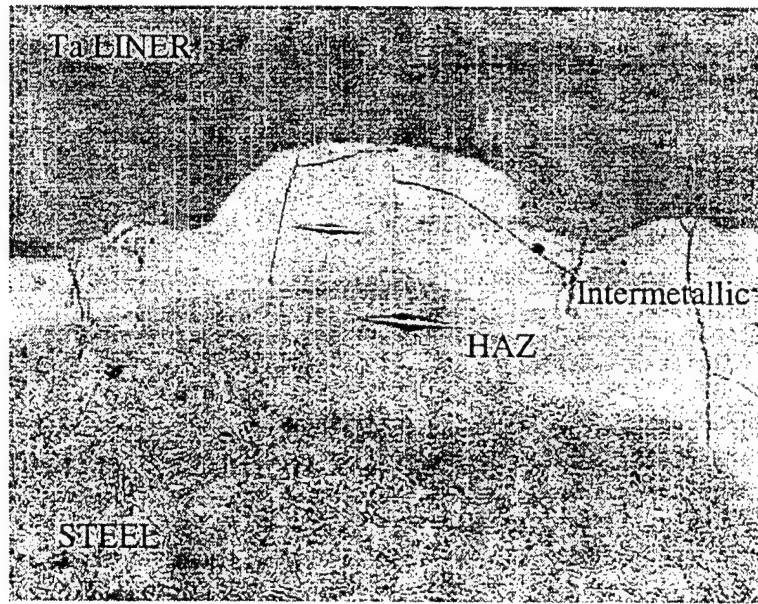


Figure 7. Optical photomicrograph showing the cracked intermetallic phase at the interface and the heat-affected zone in the steel (500X).

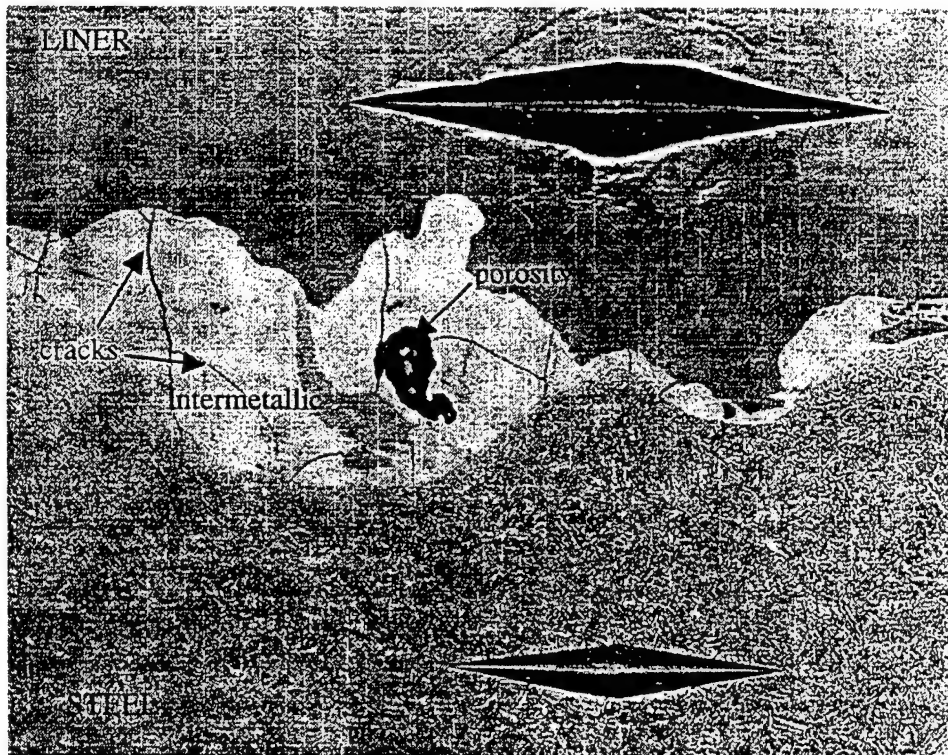


Figure 8. Optical photomicrograph showing the cracked intermetallic phase at the interface (375X). Note the porosity in the intermetallic and the difference in hardness between the coating and the steel substrate.

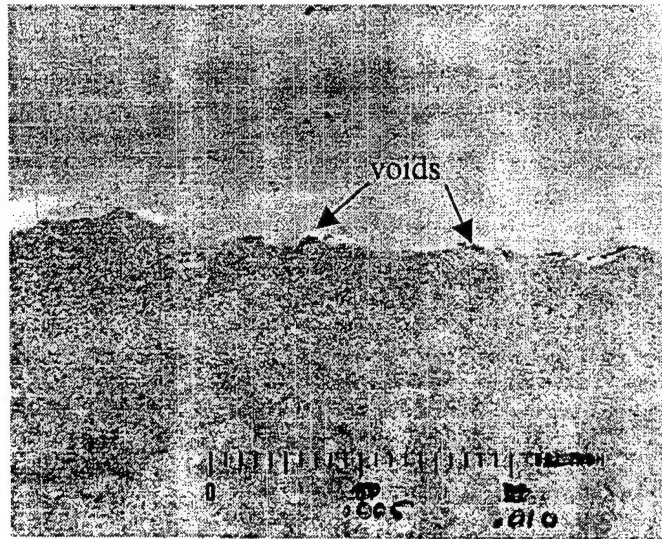


Figure 9. Optical photomicrograph showing presence of voids at the interface (200X).



Figure 10. Optical photomicrograph showing normalized microstructure (pearlite and ferrite) of the steel (500X).

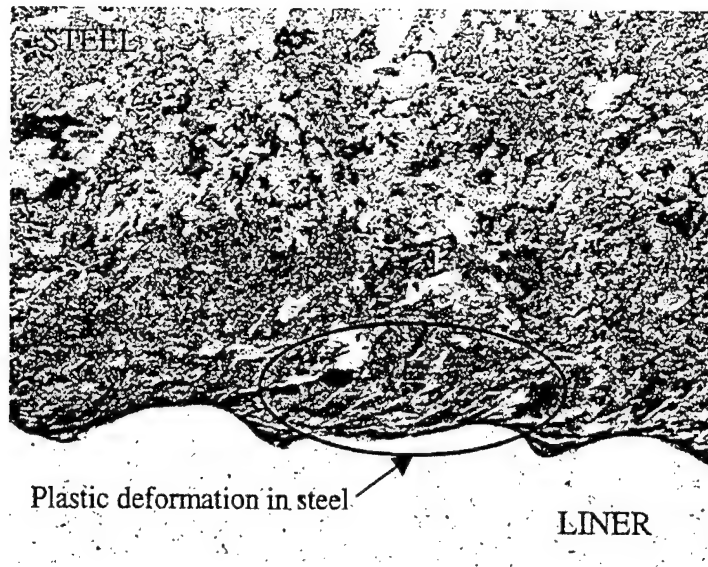


Figure 11. Photomicrograph showing deformation in the steel from the explosive bonding process (150X).

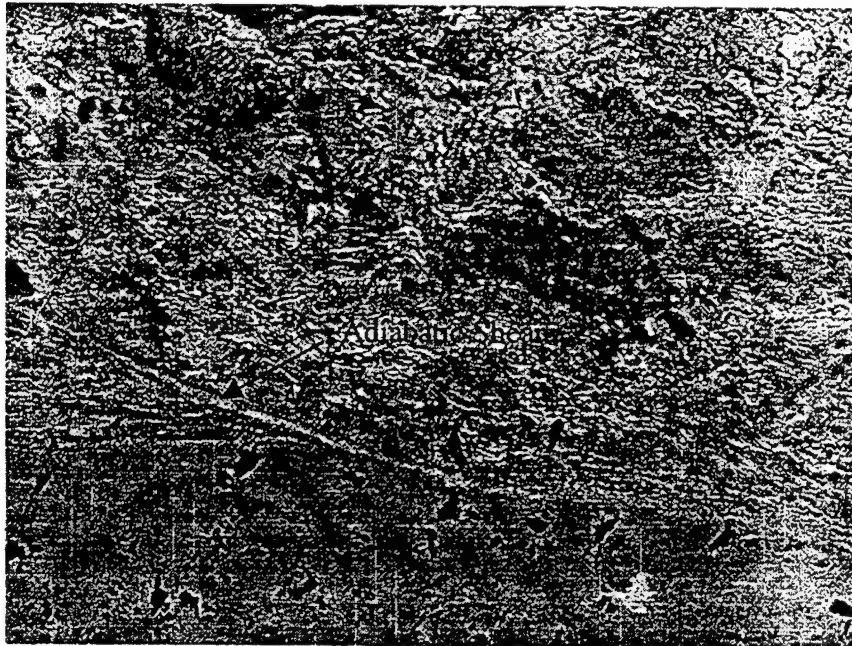


Figure 12. Photomicrograph showing adiabatic shear band in the steel (500X).

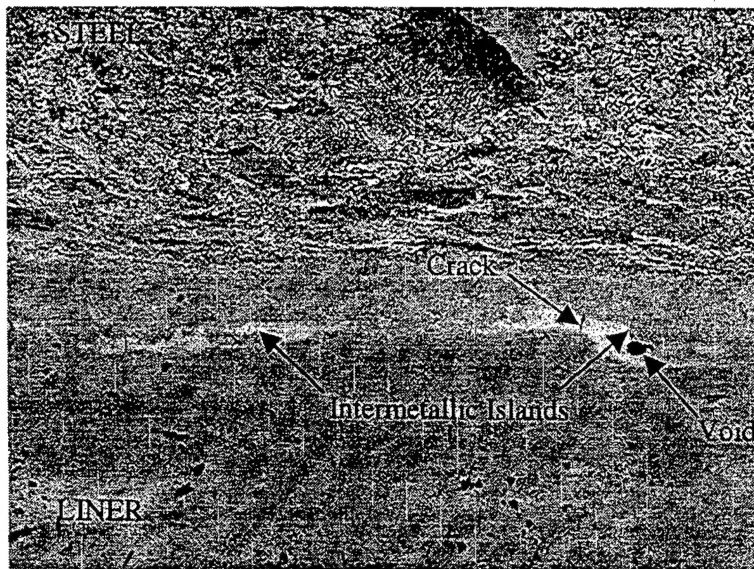


Figure 13. Photomicrograph showing the presence of voids and intermetallic "islands" in the tantalum liner (500X).

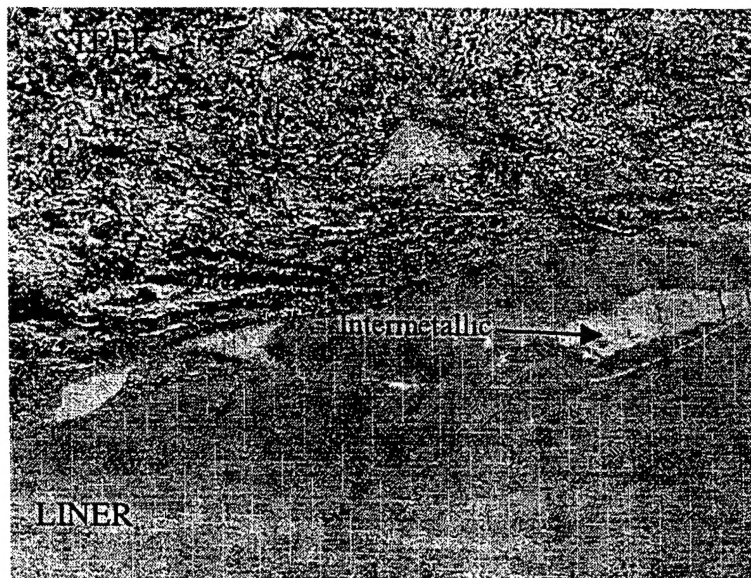


Figure 14. Photomicrograph showing crack intermetallic phase (500X).

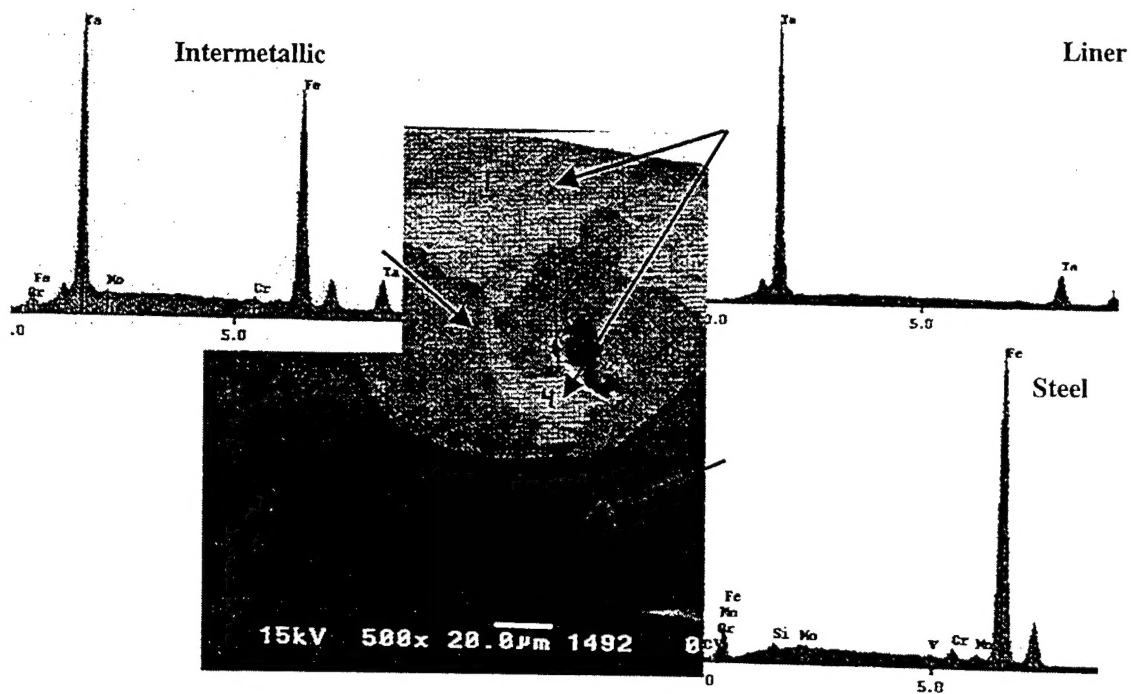


Figure 15. Results of EDS showing that the intermetallic consists of an iron/tantalum compound.

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